

1-(Carbamoylmethyl)cyclohexanecarboxylic acid

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Key indicators

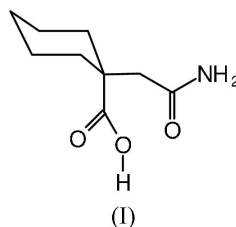
Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.038
 wR factor = 0.103
Data-to-parameter ratio = 15.1

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Molecules of the title compound, $\text{C}_9\text{H}_{15}\text{NO}_3$, form a two-dimensional hydrogen-bonded network, *via* $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ interactions, which runs parallel to the bc plane. In this structure, neither the carboxylic acid groups nor the carbamoyl groups are involved in dimer formations.

Comment

The title compound, (I), is used as an intermediate in the synthesis of biologically active heterocycles (LaRoche & Helmers, 2004). A search of the Cambridge Structural Database (Version 5.26; Allen, 2002) reveals that there are 11 structures of 1,1-disubstituted cyclohexane with a carboxylic acid group as one of the substituents. Of these, only three contain 1-cyclohexanecarboxylic acid itself. The remaining structures each contain an amino group (as the second substituent), with further attached groups on the amino N atom. There are no structures similar to 1-(carbamoylmethyl)cyclohexane.



Molecules of the title compound (Fig. 1) form a two-dimensional hydrogen-bonded network, *via* $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ interactions, which runs parallel to the bc plane. Hydrogen-bonding associations are listed in Table 1. The carboxylic OH group hydrogen bonds to the carbamoyl O atom of an adjacent molecule while the amino group of that molecule, in return, hydrogen bonds with the carbamoyl O atom of the first molecule. These two associations form a hydrogen-bonded ring motif [$R_2^2(11)$ graph set (Etter, 1990)] that, when repeated, propagates the hydrogen-bonding network in the b -axis direction. An $\text{N}-\text{H}\cdots\text{O}$ association between the second amino H atom and an adjacent carboxyl carbonyl O atom in the c -axis direction generates the two-dimensional network. Interestingly, in this structure, neither the carboxylic acid groups nor the carbamoyl groups are involved in $R_2^2(8)$ graph-set dimer formations, with like groups or with each other.

Experimental

Cyclohexanone (1.04 g, 10 mmol) was treated with ethyl cyanoacetate (1.06 g, 10 mmol) in the presence of NaOH (5 ml, 10%

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aqueous solution). The resultant compound was further treated with NaCN (0.49 g, 10 mmol) in ethanol (5 ml), and hydrolysed to obtain the title compound. Crystals were grown from methanol.

Crystal data

$C_9H_{15}NO_3$	$D_x = 1.307 \text{ Mg m}^{-3}$
$M_r = 185.22$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2268 reflections
$a = 13.4973(5) \text{ \AA}$	$\theta = 2.9\text{--}27.5^\circ$
$b = 8.0905(2) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 8.8358(3) \text{ \AA}$	$T = 120(2) \text{ K}$
$\beta = 102.627(2)^\circ$	Prism, colourless
$V = 941.53(5) \text{ \AA}^3$	$0.65 \times 0.30 \times 0.10 \text{ mm}$
$Z = 4$	

Data collection

Nonius KappaCCD diffractometer	1627 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.030$
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 2003)	$\theta_{\text{max}} = 26.0^\circ$
$T_{\text{min}} = 0.939$, $T_{\text{max}} = 0.990$	$h = -16 \rightarrow 16$
10959 measured reflections	$k = -9 \rightarrow 9$
1842 independent reflections	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.4339P]$
$R[F^2 > 2\sigma(F^2)] = 0.038$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.103$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
1842 reflections	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
122 parameters	Extinction correction: <i>SHELXL97</i>
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.048 (6)

Table 1

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O3\cdots H3\cdots O1^i$	0.963 (18)	1.640 (19)	2.5829 (13)	165 (2)
$N1\cdots H1\cdots O1^{ii}$	0.88	2.21	3.0680 (15)	164
$N1\cdots H2\cdots O2^{iii}$	0.88	2.12	2.9635 (15)	162

Symmetry codes: (i) $-x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $-x, \frac{1}{2} + y, \frac{1}{2} - z$; (iii) $x, \frac{1}{2} - y, z - \frac{1}{2}$.

The carboxyl H atom was located in a difference Fourier synthesis and its positional parameters were refined. Other H atoms were included in the refinement at calculated positions, in the riding-model approximation, with C—H distances of 0.99 \AA and N—H distances of 0.88 \AA . The isotropic displacement parameters for all H atoms were set equal to 1.25 U_{eq} of the carrier atom.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduc-

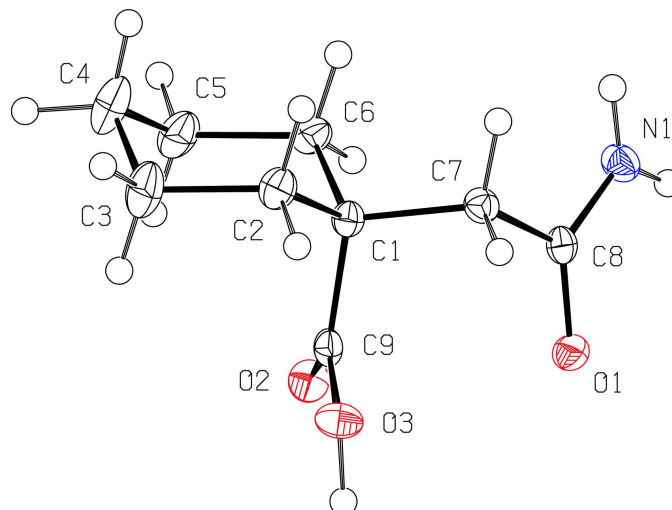


Figure 1

The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radius.

tion: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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supporting information

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Crystal data

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 $M_r = 185.22$
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 Hall symbol: $-P\ 2_1bc$
 $a = 13.4973\ (5)\ \text{\AA}$
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 $c = 8.8358\ (3)\ \text{\AA}$
 $\beta = 102.627\ (2)^\circ$
 $V = 941.53\ (5)\ \text{\AA}^3$
 $Z = 4$

$F(000) = 400$
 $D_x = 1.307\ \text{Mg m}^{-3}$
 Melting point: 437 K
 Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$
 Cell parameters from 2268 reflections
 $\theta = 2.9\text{--}27.5^\circ$
 $\mu = 0.10\ \text{mm}^{-1}$
 $T = 120\ \text{K}$
 Prism, colourless
 $0.65 \times 0.30 \times 0.10\ \text{mm}$

Data collection

Nonius KappaCCD
 diffractometer
 Radiation source: Bruker Nonius FR591
 rotating anode
 10 cm confocal mirrors monochromator
 Detector resolution: $9.091\ \text{pixels mm}^{-1}$
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2003)

$T_{\min} = 0.939$, $T_{\max} = 0.990$
 10959 measured reflections
 1842 independent reflections
 1627 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$
 $\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 3.5^\circ$
 $h = -16 \rightarrow 16$
 $k = -9 \rightarrow 9$
 $l = -10 \rightarrow 10$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.103$
 $S = 1.05$
 1842 reflections
 122 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map

Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.4339P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.20\ \text{e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.25\ \text{e \AA}^{-3}$
 Extinction correction: SHELXL97,
 $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.048 (6)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.01177 (7)	0.16623 (11)	0.19598 (11)	0.0192 (3)

O2	0.18173 (7)	−0.03683 (12)	0.38790 (11)	0.0214 (3)
O3	0.11848 (8)	−0.18613 (12)	0.17583 (11)	0.0224 (3)
H3	0.0775 (13)	−0.237 (2)	0.239 (2)	0.028*
N1	0.07782 (9)	0.40190 (14)	0.12383 (13)	0.0202 (3)
H1	0.0420	0.4629	0.1748	0.025*
H2	0.1191	0.4493	0.0724	0.025*
C1	0.22107 (9)	0.04376 (16)	0.14274 (15)	0.0157 (3)
C2	0.27157 (10)	−0.06508 (17)	0.03825 (16)	0.0207 (3)
H21	0.2217	−0.1471	−0.0157	0.026*
H22	0.2921	0.0050	−0.0415	0.026*
C3	0.36471 (11)	−0.1557 (2)	0.1304 (2)	0.0310 (4)
H31	0.3966	−0.2201	0.0585	0.039*
H32	0.3434	−0.2340	0.2033	0.039*
C4	0.44223 (11)	−0.0342 (2)	0.2213 (2)	0.0354 (4)
H41	0.4999	−0.0963	0.2844	0.044*
H42	0.4688	0.0368	0.1480	0.044*
C5	0.39417 (11)	0.0735 (2)	0.32693 (18)	0.0271 (4)
H51	0.4443	0.1561	0.3789	0.034*
H52	0.3751	0.0037	0.4081	0.034*
C6	0.29991 (10)	0.16275 (17)	0.23608 (16)	0.0199 (3)
H61	0.3207	0.2429	0.1642	0.025*
H62	0.2683	0.2255	0.3094	0.025*
C7	0.13317 (10)	0.14047 (17)	0.03674 (15)	0.0169 (3)
H71	0.1622	0.2166	−0.0298	0.021*
H72	0.0889	0.0611	−0.0320	0.021*
C8	0.06951 (9)	0.23871 (17)	0.12452 (14)	0.0159 (3)
C9	0.17348 (10)	−0.06333 (16)	0.25029 (15)	0.0161 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0178 (5)	0.0183 (5)	0.0229 (5)	0.0012 (4)	0.0078 (4)	0.0003 (4)
O2	0.0227 (5)	0.0249 (5)	0.0166 (5)	0.0010 (4)	0.0041 (4)	−0.0012 (4)
O3	0.0279 (6)	0.0187 (5)	0.0218 (5)	−0.0066 (4)	0.0077 (4)	−0.0022 (4)
N1	0.0241 (6)	0.0163 (6)	0.0219 (6)	0.0026 (5)	0.0088 (5)	0.0015 (5)
C1	0.0153 (6)	0.0155 (6)	0.0161 (7)	0.0014 (5)	0.0031 (5)	−0.0009 (5)
C2	0.0200 (7)	0.0217 (7)	0.0216 (7)	0.0030 (5)	0.0069 (6)	−0.0030 (6)
C3	0.0230 (8)	0.0316 (9)	0.0376 (9)	0.0101 (6)	0.0051 (7)	−0.0063 (7)
C4	0.0167 (7)	0.0444 (10)	0.0433 (10)	0.0075 (7)	0.0024 (7)	−0.0053 (8)
C5	0.0166 (7)	0.0317 (8)	0.0302 (8)	−0.0026 (6)	−0.0009 (6)	−0.0044 (6)
C6	0.0174 (7)	0.0201 (7)	0.0224 (7)	−0.0027 (5)	0.0046 (6)	−0.0025 (5)
C7	0.0184 (7)	0.0176 (7)	0.0151 (6)	0.0011 (5)	0.0042 (5)	0.0013 (5)
C8	0.0151 (6)	0.0181 (7)	0.0126 (6)	0.0015 (5)	−0.0009 (5)	0.0010 (5)
C9	0.0145 (6)	0.0145 (6)	0.0187 (7)	0.0038 (5)	0.0022 (5)	0.0003 (5)

Geometric parameters (Å, °)

O1—C8	1.2510 (16)	C3—C4	1.529 (2)
O2—C9	1.2156 (16)	C3—H31	0.99
O3—C9	1.3257 (16)	C3—H32	0.99
O3—H3	0.963 (18)	C4—C5	1.522 (2)
N1—C8	1.3252 (18)	C4—H41	0.99
N1—H1	0.88	C4—H42	0.99
N1—H2	0.88	C5—C6	1.529 (2)
C1—C9	1.5274 (18)	C5—H51	0.99
C1—C6	1.5348 (18)	C5—H52	0.99
C1—C2	1.5387 (18)	C6—H61	0.99
C1—C7	1.5528 (17)	C6—H62	0.99
C2—C3	1.528 (2)	C7—C8	1.5043 (18)
C2—H21	0.99	C7—H71	0.99
C2—H22	0.99	C7—H72	0.99
C9—O3—H3	111.3 (10)	C3—C4—H42	109.5
C8—N1—H1	120.0	H41—C4—H42	108.1
C8—N1—H2	120.0	C4—C5—C6	111.46 (12)
H1—N1—H2	120.0	C4—C5—H51	109.3
C9—C1—C6	110.97 (11)	C6—C5—H51	109.3
C9—C1—C2	110.51 (10)	C4—C5—H52	109.3
C6—C1—C2	109.59 (10)	C6—C5—H52	109.3
C9—C1—C7	106.94 (10)	H51—C5—H52	108.0
C6—C1—C7	110.90 (10)	C5—C6—C1	112.67 (11)
C2—C1—C7	107.86 (10)	C5—C6—H61	109.1
C3—C2—C1	112.06 (11)	C1—C6—H61	109.1
C3—C2—H21	109.2	C5—C6—H62	109.1
C1—C2—H21	109.2	C1—C6—H62	109.1
C3—C2—H22	109.2	H61—C6—H62	107.8
C1—C2—H22	109.2	C8—C7—C1	113.73 (10)
H21—C2—H22	107.9	C8—C7—H71	108.8
C2—C3—C4	111.14 (13)	C1—C7—H71	108.8
C2—C3—H31	109.4	C8—C7—H72	108.8
C4—C3—H31	109.4	C1—C7—H72	108.8
C2—C3—H32	109.4	H71—C7—H72	107.7
C4—C3—H32	109.4	O1—C8—N1	122.14 (12)
H31—C3—H32	108.0	O1—C8—C7	120.10 (12)
C5—C4—C3	110.83 (12)	N1—C8—C7	117.75 (12)
C5—C4—H41	109.5	O2—C9—O3	123.19 (12)
C3—C4—H41	109.5	O2—C9—C1	124.28 (12)
C5—C4—H42	109.5	O3—C9—C1	112.48 (11)
C9—C1—C2—C3	−67.91 (14)	C6—C1—C7—C8	−66.21 (14)
C6—C1—C2—C3	54.69 (15)	C2—C1—C7—C8	173.78 (11)
C7—C1—C2—C3	175.52 (12)	C1—C7—C8—O1	−70.50 (15)
C1—C2—C3—C4	−56.56 (17)	C1—C7—C8—N1	108.96 (13)

C2—C3—C4—C5	56.00 (18)	C6—C1—C9—O2	13.30 (17)
C3—C4—C5—C6	−55.11 (18)	C2—C1—C9—O2	135.09 (13)
C4—C5—C6—C1	55.11 (16)	C7—C1—C9—O2	−107.76 (14)
C9—C1—C6—C5	68.36 (14)	C6—C1—C9—O3	−169.07 (11)
C2—C1—C6—C5	−53.96 (15)	C2—C1—C9—O3	−47.28 (14)
C7—C1—C6—C5	−172.93 (11)	C7—C1—C9—O3	69.87 (13)
C9—C1—C7—C8	54.90 (14)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O3—H3 \cdots O1 ⁱ	0.963 (18)	1.640 (19)	2.5829 (13)	165 (2)
N1—H1 \cdots O1 ⁱⁱ	0.88	2.21	3.0680 (15)	164
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